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10 - 22°K CRYOSORPTION OF HELIUM ON MOLECULAR SIEVE 5A AND HYDROGEN ON CONDENSED VAPORS

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R. E. Southerlan
ARO, Inc.

May 1965

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**AEROSPACE ENVIRONMENTAL FACILITY
ARNOLD ENGINEERING DEVELOPMENT CENTER
AIR FORCE SYSTEMS COMMAND
ARNOLD AIR FORCE STATION, TENNESSEE**

10 - 22°K CRYOSORPTION OF HELIUM
ON MOLECULAR SIEVE 5A
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R. E. Southerlan
ARO, Inc.

FOREWORD

The results of research reported herein were obtained by ARO, Inc. (a subsidiary of Sverdrup and Parcel, Inc.), contract operator of the Arnold Engineering Development Center (AEDC), Air Force Systems Command (AFSC), Arnold Air Force Station, Tennessee, under Contract AF 40(600)-1000, Program Element 65402234/7778, Task 777801. The research was conducted from February to June 1964 under ARO Project No. SN2304, and the report was submitted by the author on February 11, 1965.

This technical report has been reviewed and is approved.

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ABSTRACT

Experimental results are presented for helium adsorption on molecular sieve 5A at 10 and 13.6°K. Results are also given which show that water frost near 22°K adsorbs hydrogen at high volumetric rates with significant adsorptive capacity. Carbon dioxide frosts demonstrated these characteristics to a lesser degree near 16°K.

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SECTION I INTRODUCTION

Production of high and ultrahigh clean vacuums has become a significant prerequisite to space environmental testing. Cryogenic pumping is now a widely accepted technique for gas removal; however, hydrogen, helium, and neon cannot be economically pumped by this method. In practice neon is seldom considered by virtue of its rarity, and diffusion pumping is accepted as best applicable for hydrogen and helium removal.

Diffusion pumping poses a series of application compromises. Limited amounts of wall space are usually available for mounting. Cleanliness required by optical systems and test vehicles demands rather elaborate oil trapping, imposing additional conductance restrictions. In contrast, recently developed cryosorption materials and techniques (Refs. 1, 2, and 3) eliminate contamination problems and can be used to provide high pumping speeds for both hydrogen and helium.

Inherent characteristics minimize to a degree some advantages realized by cryosorption addition. Of primary concern is the fact that the materials used have a finite loading capacity. When this capacity is realized, the pump is no longer of value until a reactivation is accomplished-- at the compromise of the attained vacuum. With this in mind, the concept of continuous deposition of an adsorbent, particularly for large hydrogen gas loads, is attractive. Titanium gettering employs this approach but does not utilize the advantages of 20°K cryogenics.

Earlier work (Refs. 1, 3, 4, and 5) had been conducted to develop and evaluate the cryosorption material known as molecular sieve 5A. Programs at AEDC demonstrated the effectiveness of this material for hydrogen removal with near 20°K sieve temperatures. A barely measurable helium pumping speed was observed using 17°K sieve temperatures which encouraged further study using a lower temperature refrigerant.

Results obtained in the earlier work at AEDC defined additional areas of interest. In an attempt to contaminate the molecular sieve adsorbent, it was found that water vapor, when applied as a frost, exhibited high hydrogen pumping speeds. Additional work was initiated to determine water frost properties when deposited on stainless steel panels.

The attractiveness of vapor adsorbents having lower triple points than water (H_2O) and exhibiting the desired properties is apparent. Ease of application in the presence of a surrounding 77°K environment is of prime concern. Inasmuch as Hemstreet, et al. (Ref. 2) reported some success in adsorbing hydrogen on carbon dioxide frosts, a portion of the study was devoted to further evaluating this material.

SECTION II TEST HARDWARE

All testing was accomplished in the Aerospace Research Chamber (7V) (Fig. 1). The basic chamber configuration consists of the following:

- (1) A cylindrical, sandblasted, stainless steel chamber seven feet in diameter and twelve feet in length.
- (2) An interior liquid-nitrogen-cooled cylindrical liner and end panels reducing the cylindrical working dimensions to 6.25 by 12 ft.
- (3) Two 32-in. oil diffusion pumps baffled by optically dense liquid-nitrogen-cooled chevrons.
- (4) Two 6-in. oil diffusion pumps with 6-in. slide valves in series with the 32-in. pumps.

Refrigerant from 13 to 22°K was provided by a 1-kw helium refrigeration unit. Various cryopump configurations were employed which will be described later. Near 10°K panel temperatures were achieved by using liquid helium supplied from 100-l Dewars.

A 300-g molecular sieve 5A sample was used in the study (Fig. 2). An embossed stainless steel panel served as the sieve substrate and refrigerant carrier. Installation of activation heaters and refrigerant plumbing completed the panel installation.

Panel dimensions were 29 by 47 in. with a light-gage sheet metal binding edge limiting the effective exposed sieve area to 8.35 ft² (Fig. 2). A 16- by 16-mesh screen fabricated of 0.019-in.-diam stainless steel wire was used as a supporting lath for the adsorbent. Intermittent brazing of mesh to substrate provided additional mechanical strength and conductive cooling. Portions of this mesh were visible on the surface, resulting in a small percentage reduction in the effective pumping area. Actual sieve thickness was not constant over the entire panel surface, but varied near a mean thickness of 1/16 in.

Sieve activation was accomplished by resistive heating. Six 500-w strip heaters, controlled by rheostats and mounted to the substrate panel (Fig. 2), provided activation temperature levels of 500°F.

2.1 INSTRUMENTATION

The test gas admission system is shown in Fig. 3. Two constant conductance leaks made from porous sintered stainless steel plugs metered the gas (Ref. 6). Periodic calibration throughout the test confirmed constant conductance values of the leaks. Chamber pressures were measured by nude ionization gages and a mass spectrometer.

Six copper-constantan thermocouples were used to monitor panel activation temperatures. Cryogenic operating temperatures were determined by a hydrogen vapor pressure thermometer mounted in the appropriate cryosorption panel outlet.

2.2 VACUUM GAGE CALIBRATION

Nude gage and mass spectrometer sensitivities were determined in-chamber with 77°K helium and 300°K hydrogen. For helium calibrations the cell was evacuated to base pressure with a 27.7-sq ft, 17°K cryosurface and two 6-in. diffusion pumps. Small, known volumes were filled with the test gas and the corresponding charge pressure was determined by a pressure transducer. No detectable helium leak was observed, and no increase in pressure was experienced after the 6-in. pumps were isolated. The small volumes were then expanded into the known cell volume to provide known pressure increments for gage comparison. Repeated gas admissions were made using the various volumes and charge pressures, and highly repeatable results were obtained over several orders of magnitude in pressure.

This success was far from realized in the hydrogen calibrations. The same technique was employed, but a small residual hydrogen pressure was noted. Hydrogen gas admission from the known volumes resulted in an instantaneous pressure increase followed by a rapid reduction in pressure to values observed before gas admission. It was concluded that cryopumped water vapor originating from cell outgassing during the early pumpdown stages and condensed on the 20°K surface provided the observed pumping capacity.

The technique proved successful when accomplished without 20°K cryosurfaces. Base pressures were higher, and a hydrogen partial

pressure increase was observed upon closing the diffusion pumps. This resulted in a sacrifice of calibration range at low pressures, and linear characteristics of the instrumentation below 10^{-5} torr were relied on for the low pressure measurements.

SECTION III TEST PHASES - CELL CONFIGURATIONS AND PROCEDURES

3.1 PHASE I - 77°K HELIUM ADSORPTION WITH 13.6°K MOLECULAR SIEVE 5A

A chamber-within-a-chamber technique was employed in this phase, thereby shielding the test section from gas contamination originating from wall outgassing. A copper cryopanel cooled to 20°K (Fig. 4) was used as a pump exposed to both the test section and annular section between the chamber and liquid-nitrogen-cooled liner.

Low temperature gaseous helium refrigerant entered the cryosorption panel inlet and exhausted through the copper cryopanel as shown schematically in Fig. 5. Refrigeration capacity dictated a low temperature operating limit of 13.6°K measured at the cryosorption panel outlet.

Previously administered gas loads were desorbed from the molecular sieve 5A by a high temperature activation schedule shown in Fig. 6. During this activation, the 6-in. diffusion pumps were operated, the liner was cooled to LN₂ temperatures, and a 2-ft-diam externally operated valve in one end panel was opened to provide additional conductance to the pumps. Prior to gas admission, the panel was cooled to operating temperatures, the 6-in. pumps were isolated, and the 2-ft separating valve was closed.

3.2 PHASE II - HYDROGEN ADSORPTION WITH WATER FROSTS

Phase II installation is shown in Fig. 7. To facilitate proper water frost location, only the gaseous-helium-cooled surfaces were refrigerated. Exposed cryosurface totalled 27.7 ft². Panel outlet temperatures were monitored and, because of the additional heat load imposed by 300°K radiation, 2.5°K differentials existed between the panel inlet and outlet.

Water vapor was introduced directly below the cryopanel from an externally heated flask. No attempt was made to meter the flow, the amount introduced being determined by a weight measurement. The

boiling rates were kept low, and the frost was applied at approximately 1.0×10^{-5} torr. In both the 12.5- and 100-g adsorbent runs, a clearly visible frost was formed.

3.3 PHASE III - HYDROGEN ADSORPTION ON CARBON DIOXIDE FROST

The operating procedure was the same as that followed during the water frost runs. A steady flow rate of carbon dioxide (CO₂), the adsorbent gas, was maintained. This yielded a constant cell pressure and permitted an accurate determination of the amount of CO₂ deposited. A total mass deposit of 22.4 g was used as the adsorbent. Following completion of the CO₂ deposition all liquid nitrogen surfaces were refrigerated.

3.4 HELIUM ADSORPTION WITH NEAR 10°K MOLECULAR SIEVE 5A

The test configuration used for this phase is shown in Fig. 7. During the test run, the 32-in. pumps were inoperative and the 6-in. pumps were valved off. Additional pumping for 20°K condensable gases was provided by operating the 27.7-ft² cryosurface.

Test preparation was essentially the same as described in section 3.1. All liquid nitrogen surfaces were cooled, and a panel activation was performed. To conserve the liquid helium supply, following activation, the panel was allowed to radiatively cool several hours to approximately 100°K before helium coolant was admitted. As each 100-l Dewar was expended, approximately five minutes were required to manually change out Dewars and reestablish steady refrigerant flow conditions.

SECTION IV EXPERIMENTAL APPROACH

The property common to all adsorbents was the volumetric pumping speed. In most applications the performance degradation of volumetric pumping speed as a function of the amount of preadsorbed gas poses a difficult phenomenon to describe because the pumping speed is generally transient. Equilibrium capacity measurements are of significant value, but little can be inferred from these regarding pumping speeds. In this experiment constant leak-corresponding pressure measurements were used to define volumetric pumping speed over the desired pressure range.

The relation governing volumetric pumping reduces from the perfect gas law and the conservation of mass to

$$\frac{Q}{P_p - P_B} = S_p$$

where

Q is the leak in torr ℓ/sec

P_p is the pumping pressure in torr (Fig. 8)

S_p is the pumping speed in ℓ/sec

P_B is the base pressure attainable with no gas inbleed and in adsorption is highly dependent on the preadsorbed gas amount

For many adsorbent test gas combinations, steady-state measurements of pumping speed could be made. For the transient case as shown in Fig. 8, an indicated pumping speed was determined based on the pressure at which the pressure-time curve slope became constant after the initial transient had subsided. In all cases the continued transient represented less than 5 percent of the test gas flow rate; conversely, at least 95 percent of the metered flow was being adsorbed. The base pressure (P_B), with the exception of hydrogen adsorption on CO_2 frost, was negligible as compared with the pumping pressure, P_p .

SECTION V RESULTS

The following pumping speed measurements were made with the test conditions shown.

Adsorbent	Temperature, °K	Gas Adsorbed	Area, ft^2	Maximum Speed, ℓ/sec
Molecular Sieve 5A	13.6	77° Helium	8.35	460
Molecular Sieve 5A	10.0	77° Helium	8.35	6000
Water Frost	21.3	300° Hydrogen	27.7	1.0×10^6
Carbon Dioxide	16.3	77° Hydrogen	27.7	2.0×10^4
Carbon Dioxide	15.3	77° Hydrogen	27.7	3.0×10^5

5.1 77°K HELIUM ADSORPTION ON 13.6°K MOLECULAR SIEVE 5A

Data presented in Fig. 9 depict helium pumping speed with molecular sieve 5A. Prior to this run, the panel had been subjected to five repeated activations, all under continuous vacuum (Ref. 3). All data were taken with only cryogenic pumps operative. Pumping speeds of 460 l/sec were obtained with some decrease noted with increasing test gas flow rates, similarly observed in other work at AEDC (Ref. 3) using hydrogen as the test gas.

5.2 300°K HYDROGEN ADSORPTION WITH WATER FROST

Pumping speed data taken on 12.5- and 100-g water frost samples are shown in Fig. 10. Averaging both sets of data, a capture coefficient of 0.75 for the thick frosts was determined, with better uniformity in the data obtained with the larger amount of water deposited.

Isotherm data for the two samples (Fig. 11) indicated that the total capacity for both deposits was essentially the same. After acquisition of the 12.5-g capacity data, hydrogen gas was admitted to the chamber until a residual or base pressure of 1.0×10^{-3} torr was observed. The addition of 2.5 g of water resulted in a pressure decrease to 2.0×10^{-6} torr and a hydrogen removal of 25 atm cc.

5.3 77°K HYDROGEN ADSORPTION ON CARBON DIOXIDE FROST

A significant increase in background hydrogen pressure was noted during the 22.4-g CO₂ deposition, resulting from hydrogen contained in the CO₂ supply. All liquid nitrogen surfaces were then cooled, and adsorbent cooling was continued to 15.3°K to achieve the lowest cell pressure (mid 10⁻¹⁰). Mass spectrometer measurements indicated that cell pressure resulted almost entirely from hydrogen, and continued to decrease with decreasing panel outlet temperatures. An almost immeasurable panel outlet temperature decrease from 15.3°K caused a cell pressure drop from low 10⁻⁹ to mid 10⁻¹⁰ torr.

Considering the increase in hydrogen background during the CO₂ deposition, it was felt that a significant amount of adsorbed hydrogen could have been contained in the initial deposit. Logically, it was assumed that warming the CO₂ deposit would liberate any adsorbed hydrogen. This was found to be true since hydrogen pressure increased with increasing panel temperatures with the 6-in. diffusion pumps operating. At approximately 20°K, the hydrogen partial pressure started a sustained decrease with continued panel warming. The temperature increase was continued until

the nitrogen partial pressure became significant, occurring near 28°K. Recooling the panel to desired operating temperature and valving off the diffusion pumps preceded the test run.

No measurable hydrogen pumping speed was observed with a 22°K panel outlet temperature with no diffusion pumps operating. A momentary pumping speed of $1.9 \times 10^4 \text{ l/sec}$ shown in Fig. 12 was measured when a 16.3°K panel outlet was maintained. After a short steady-state pumping period, the cell pressure began to increase. The remaining speeds were calculated based on the instantaneous pressure observed while continuing the constant inbleed rate. Recalling earlier discussion, section 4.0, the transient cell pressure rate of rise was caused by less than 5 percent of the flow.

Cooldown to 15.3°K was effected and the previously terminated hydrogen inflow restarted. A momentary steady-state pumping speed was again observed but was over an order of magnitude higher than observed with a 16.3°K panel outlet; this was followed by a rapid decrease as the adsorbent was loaded. Flow was then terminated and the cell was allowed to pump down. A hydrogen flow five times the previous flow was started. A three-fold pumping speed increase was noted, indicating a diffusion possibly combined with some other rate limited process. After a total cumulative loading of 165 atm cc, the inbleed was again terminated and the cell allowed to pump down. Some pumping speed recovery was observed when the larger flow of 0.43 torr l/sec was initiated, followed by the decrease shown in Fig. 12.

A measure of CO₂ frost capacity for a range of temperatures is shown in Fig. 13. As compared to water frosts, the CO₂ frost capacity was small, even at lower temperature.

5.4 77°K HELIUM ADSORPTION WITH NEAR 10°K MOLECULAR SIEVE 5A

Following an activation and panel cooldown to 100°K, helium from the liquid storage Dewars was admitted. Only cryogenic pumping was used during the test.

Figure 14 displays helium pumping speed versus pumping pressure for panel outlet temperatures near 10°K. The pumping speed decrease with increasing flow rate and/or mass loading was again observed. All pumping speed measurements were made at steady-state pressures.

No true isotherm data were obtained since flow control was not sufficiently sensitive to establish controlled temperatures. However, Fig. 15 depicts a pressure-temperature relation with 8.02 atm cc in the

closed cell volume. It should be pointed out that 2 atm cc of free helium are required for a cell pressure of 9.0×10^{-5} torr. As the curve in Fig. 15 implies, the capacity of molecular sieve 5A for helium near 10°K is highly dependent on adsorbent temperature; a 1°K reduction in temperature resulted in a ten-fold reduction in cell pressure.

SECTION VI DISCUSSION

The results indicate that molecular sieve 5A will adsorb helium in small quantities at temperatures within the capability of gaseous helium refrigeration systems. Higher pumping speeds were observed as adsorbent temperatures were decreased, particularly for small test gas flows. As shown in Fig. 14, helium could be pumped with 8.35 sq ft of adsorbent area at speeds comparable to a baffled 16-in. oil diffusion pump. A further reduction in temperature should result in an increase in pumping speed as indicated by Figs. 9 and 14.

Even with the limited pumping speed at 13.6°K, molecular sieve 5A has advantages. Modern leak detection and construction techniques can ensure that intermediate-size chambers such as was used in this study have helium leakages less than 10^{-7} torr l/sec. Thus, an adsorbent amount such as used would initially provide a helium background of less than 1.0×10^{-11} torr. Pumping speed and capacity data in Fig. 9 indicate that, for such a system leakage, helium partial pressures below 10^{-8} torr could be maintained for a year.

Earlier work at AEDC confirmed the hydrogen adsorption properties of molecular sieve 5A. However, hydrogen pumping speeds of water frost were generally higher than those determined for molecular sieve 5A despite higher adsorbent temperatures.

The success in reducing hydrogen pressure by adding 2.5 g of water frost suggests several techniques for using water frost. For small hydrogen gas loads, such as outgassing from ambient temperature steels and small hydrogen leaks, a batch deposit of 0.5 g/ft² on 20°K surfaces equaling 20 percent of the wall area would limit hydrogen pressure to below 10^{-8} torr for several days. If the hydrogen pressure became excessive a redeposition of water frost could be used to restore the pumping capability. No data were taken in this study concerning simultaneous H₂O and H₂ pumping on 20°K surfaces, but, intuitively, one would assume that continuous deposition of water vapor would satisfy high hydrogen flow rate pumping requirements.

Although previous research (Ref. 2) indicated that CO₂ would adsorb hydrogen, it was found in this study that sub-20°K adsorbent temperatures should be used. From Fig. 12 it appears that approximately 15°K is a significant temperature when CO₂ frosts are used as adsorbents.

The adsorption mechanics of CO₂ apparently differed from other adsorbents. This was indicated by the behavior of the residual pressure with large amounts of gas adsorbed after test gas admission. As shown in Fig. 16, residual pressures became increasingly larger percentages of the pumping pressure as the mass loading increased. Figure 16 shows that long-term diffusion effects influenced the data and that a residual pressure (P_b) suitable for use in the defining pumping speed equation could not be obtained. Therefore, the pumping speed data presented indicate instantaneous pumping speeds based on the observed pumping pressure under flow conditions, neglecting any effects of a base pressure (P_b).

SECTION VII **CONCLUSIONS**

Cryosorption techniques can be very useful in removing hydrogen and helium from vacuum systems. The following conclusions can be determined from the experiments:

1. Molecular sieve 5A will adsorb helium, but sieve temperatures are important.
2. Hydrogen gas can be efficiently removed with common vapor frosts near 20°K.
3. Thin water frosts exhibit adsorbent capacities exceeding 10 atm cc/g in high vacuum.
4. Carbon dioxide exhibits cryosorption properties to a lesser degree than water with operating temperatures near 16°K.
5. Adsorbent redeposition with water or carbon dioxide provides a means for continuous capacity.

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ULTIMATE PRESSURE - 1×10^{-10}

He REFRIG. SYSTEM - 1KW AT 20°K

LN₂ REFRIG. SYSTEM -

50KW AT 100°K

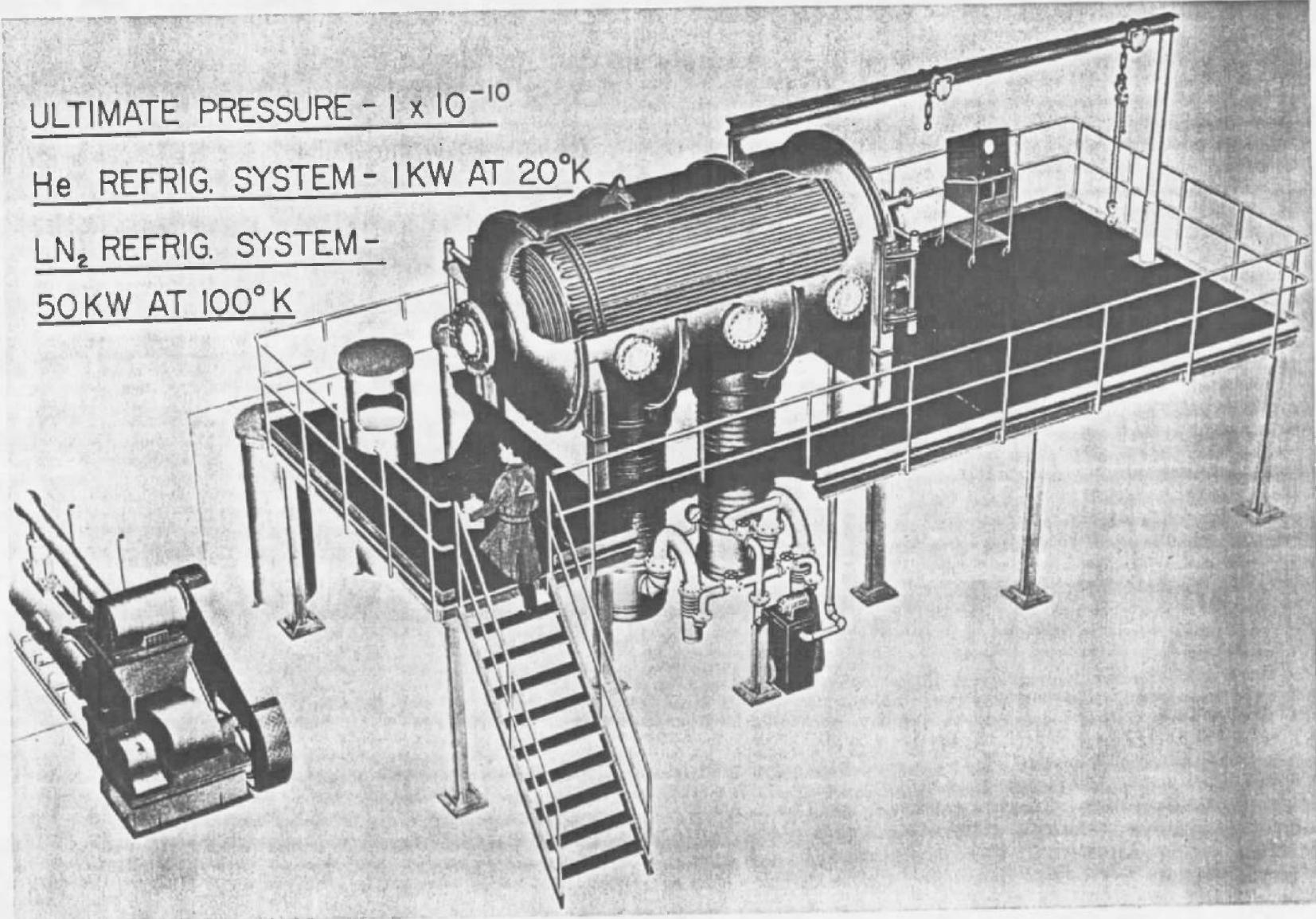


Fig. 1 Aerospace Research Chamber (7V)

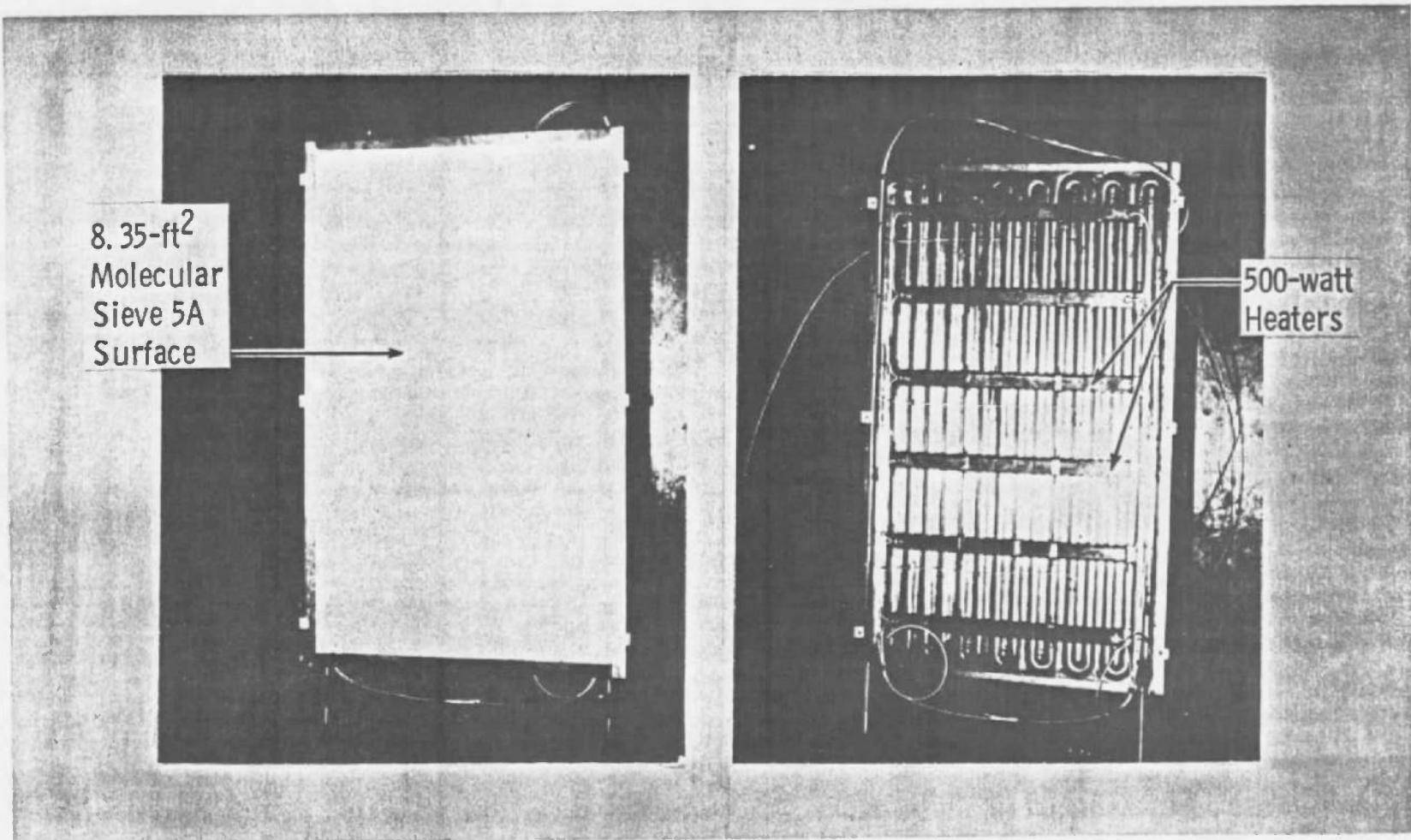


Fig. 2 Adsorbent Panel and Activation Heater Installation

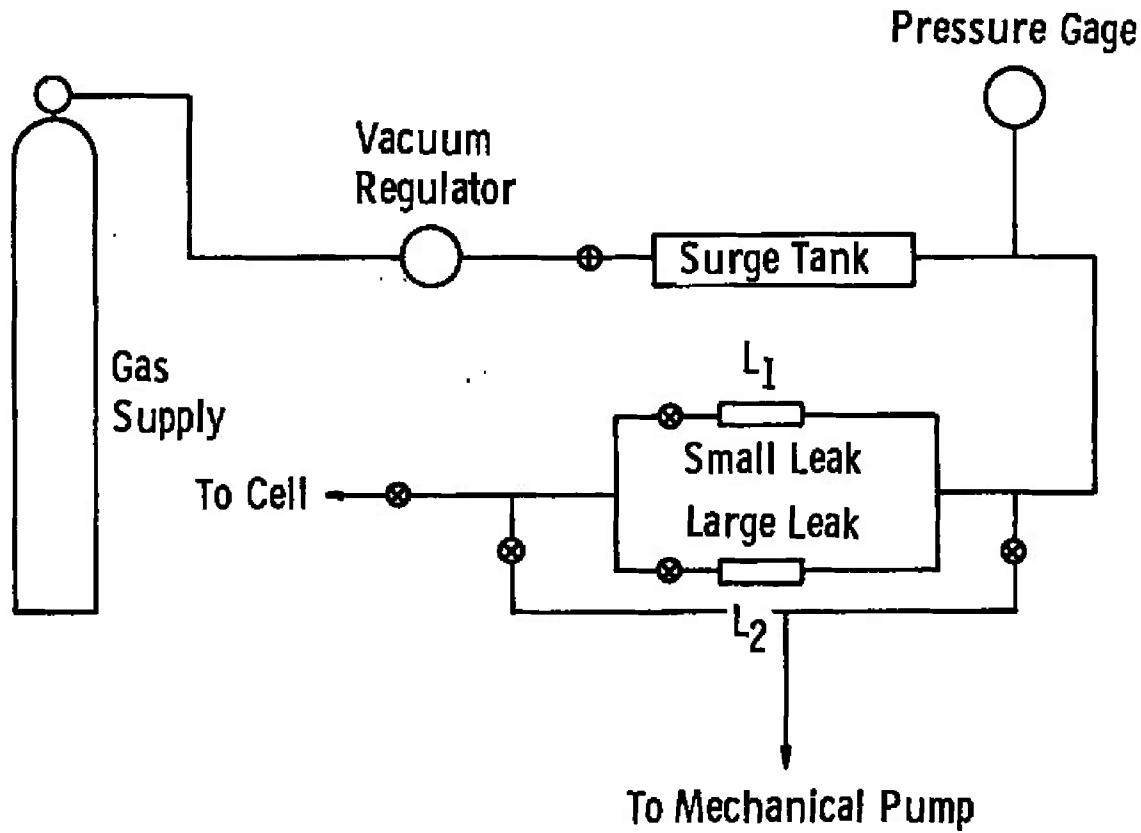


Fig. 3 Gas Inbleed System

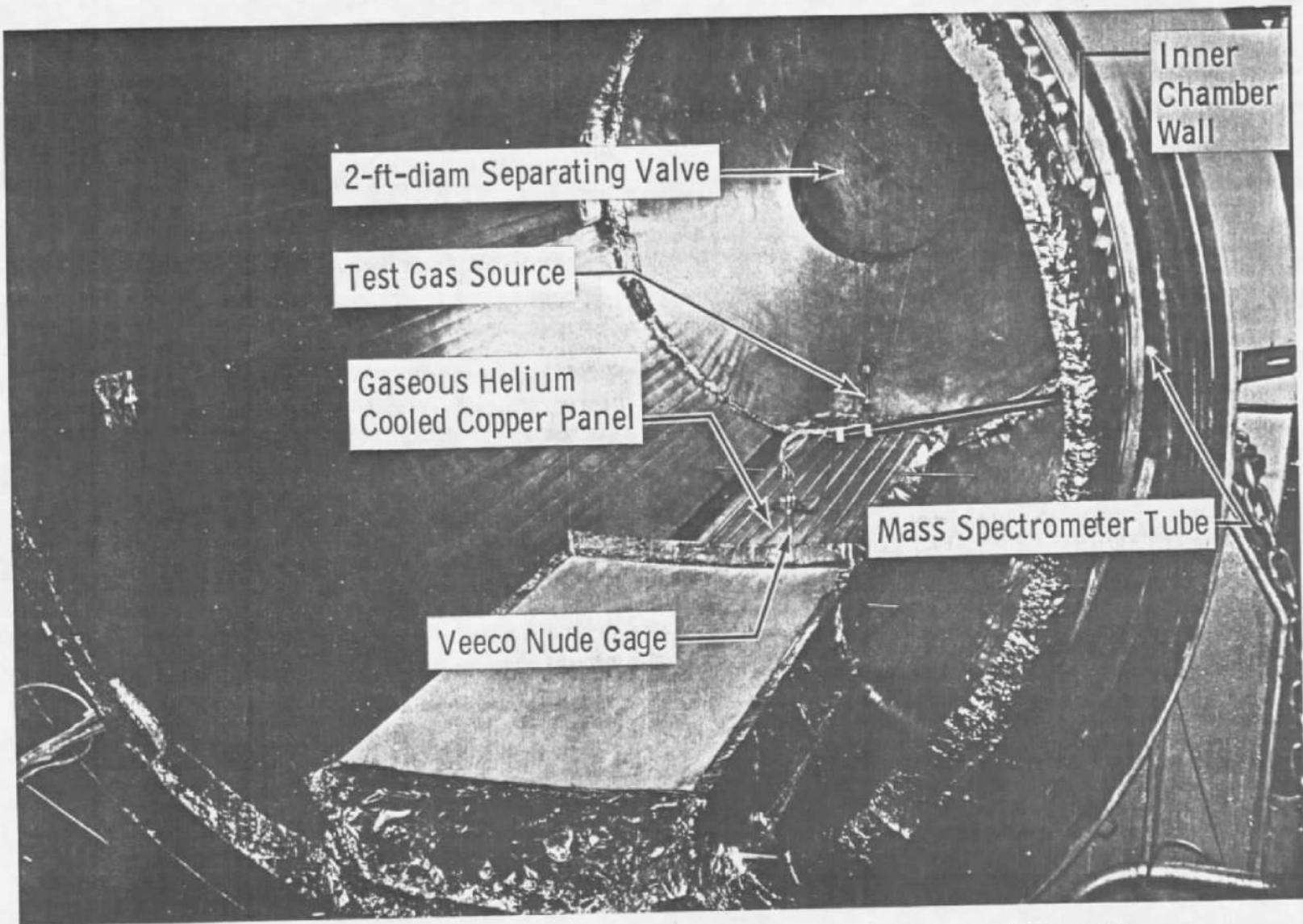


Fig. 4 Test Configuration for Hydrogen and Helium Adsorption with Molecular Sieve 5A Near 20°K

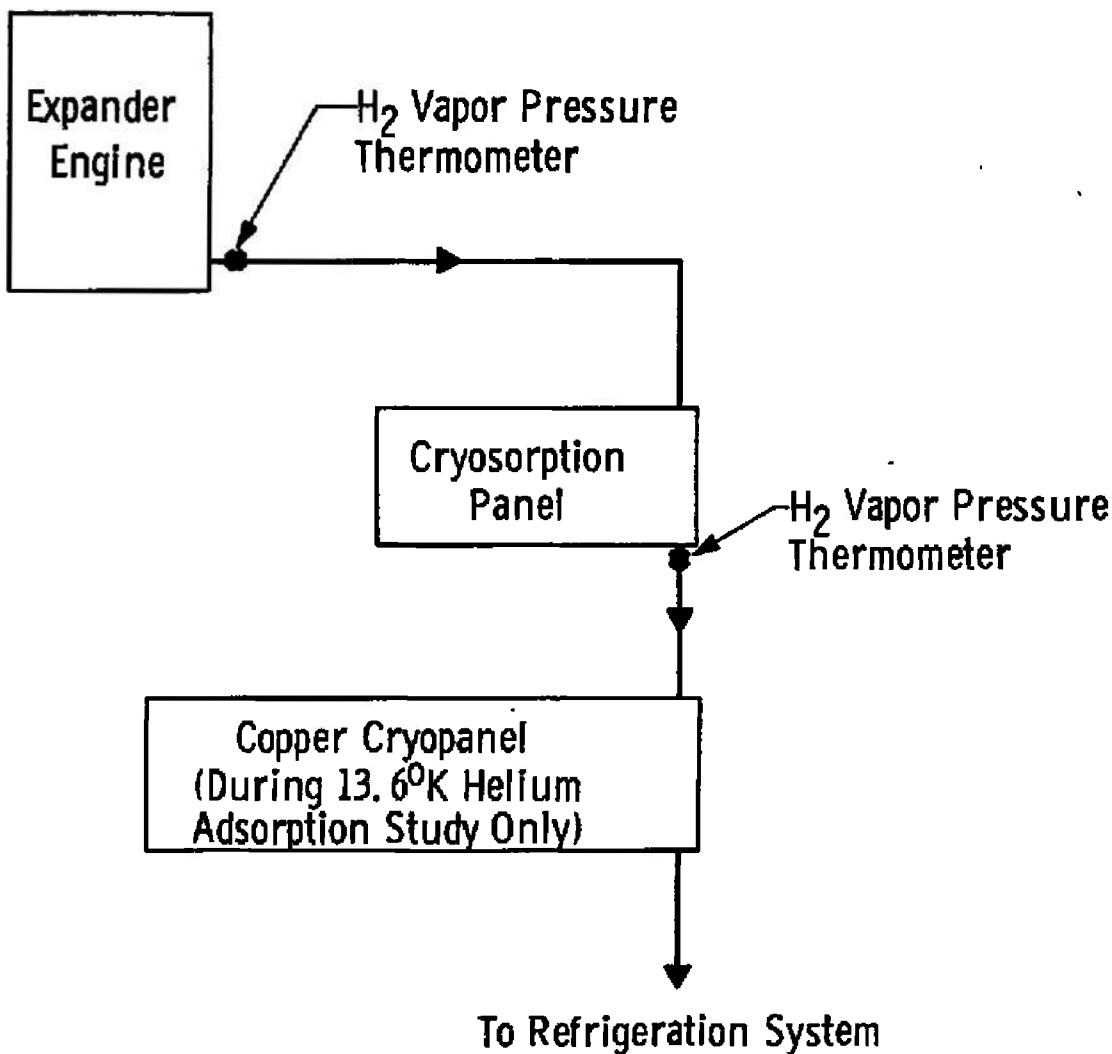


Fig. 5 Gaseous Helium Refrigerant Flow Diagram

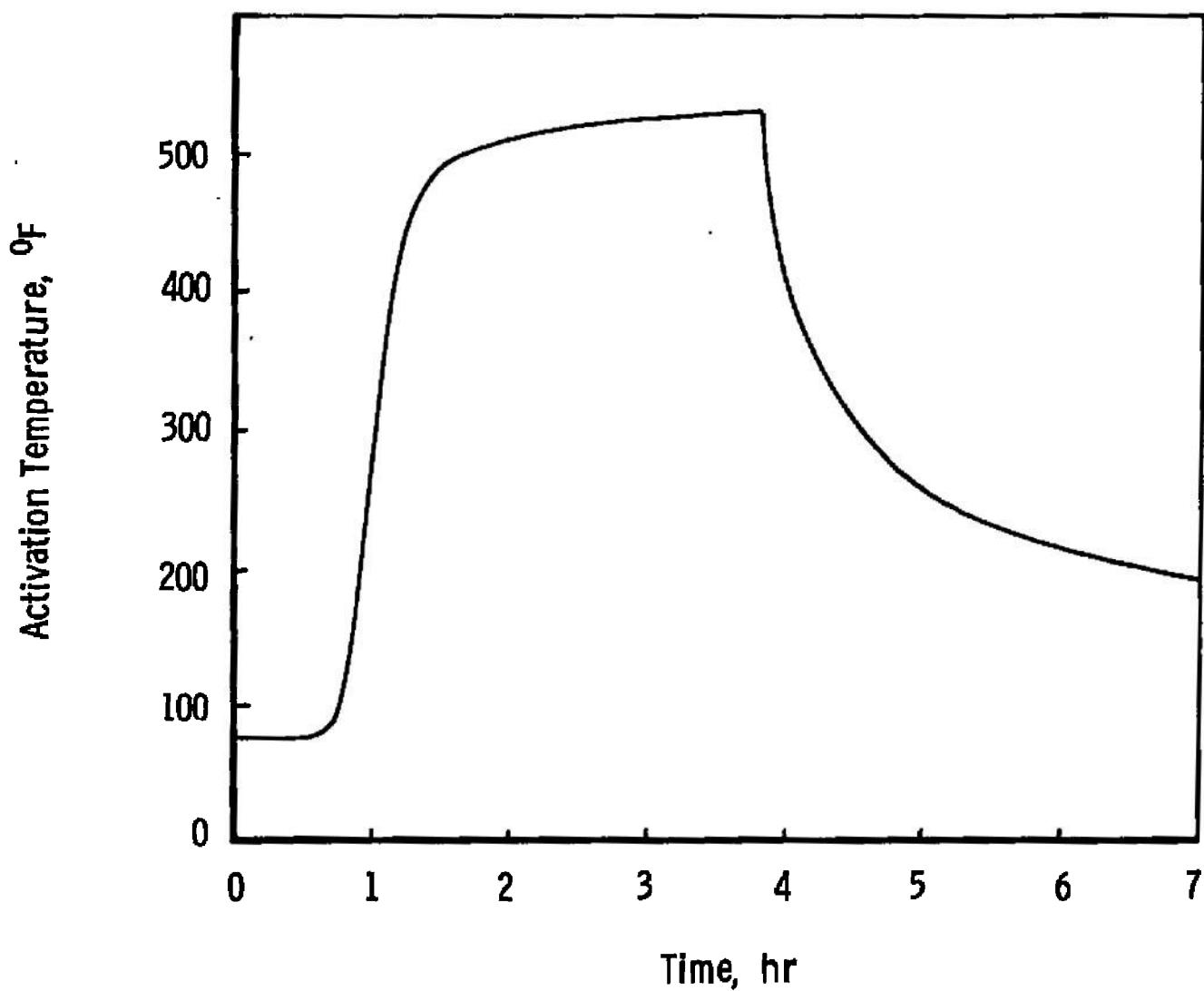


Fig. 6 Typical Panel Activation Schedule

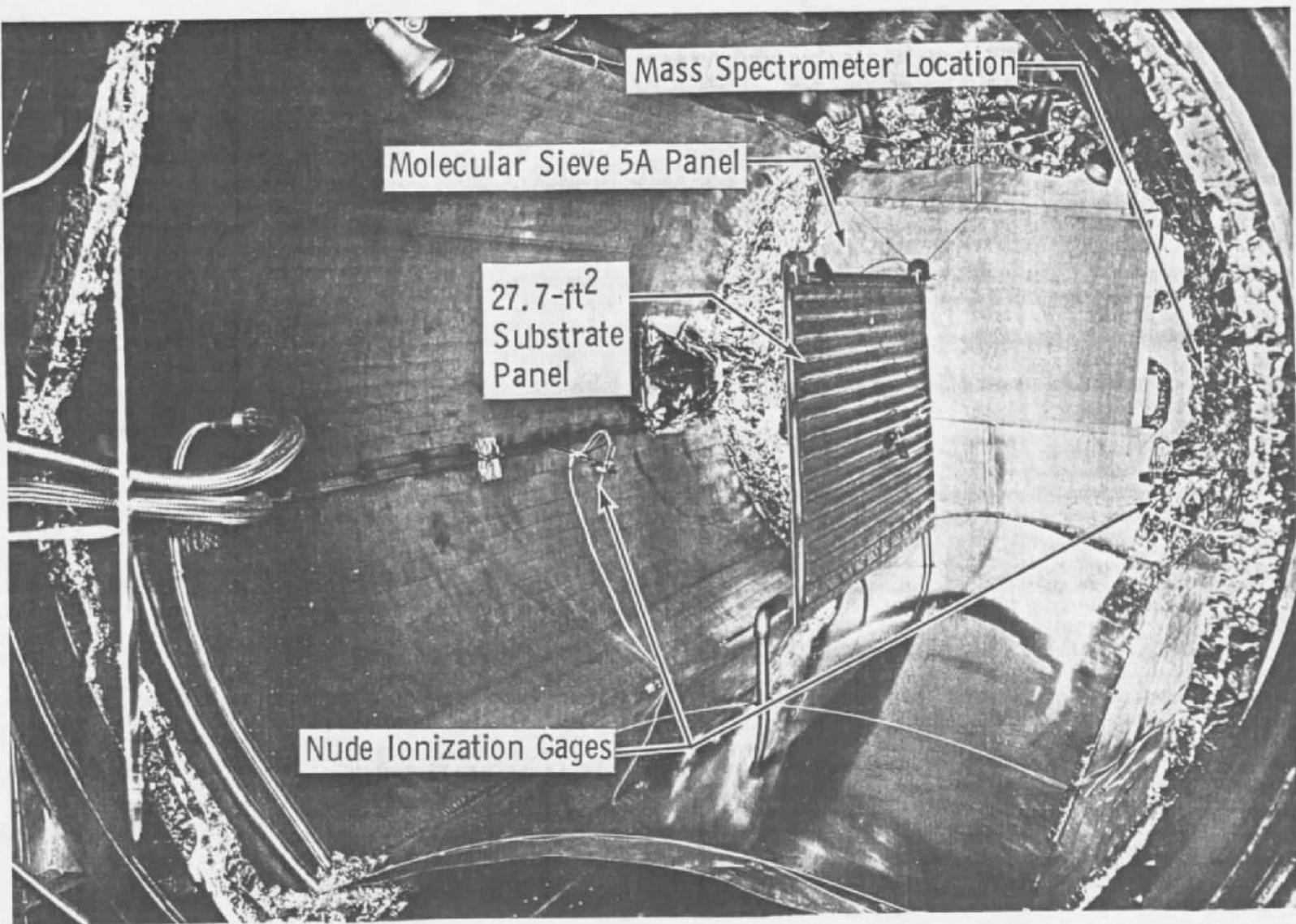


Fig. 7 Installation for Liquid-Helium-Cooled Run, Water Vapor and Carbon Dioxide Adsorbent Runs

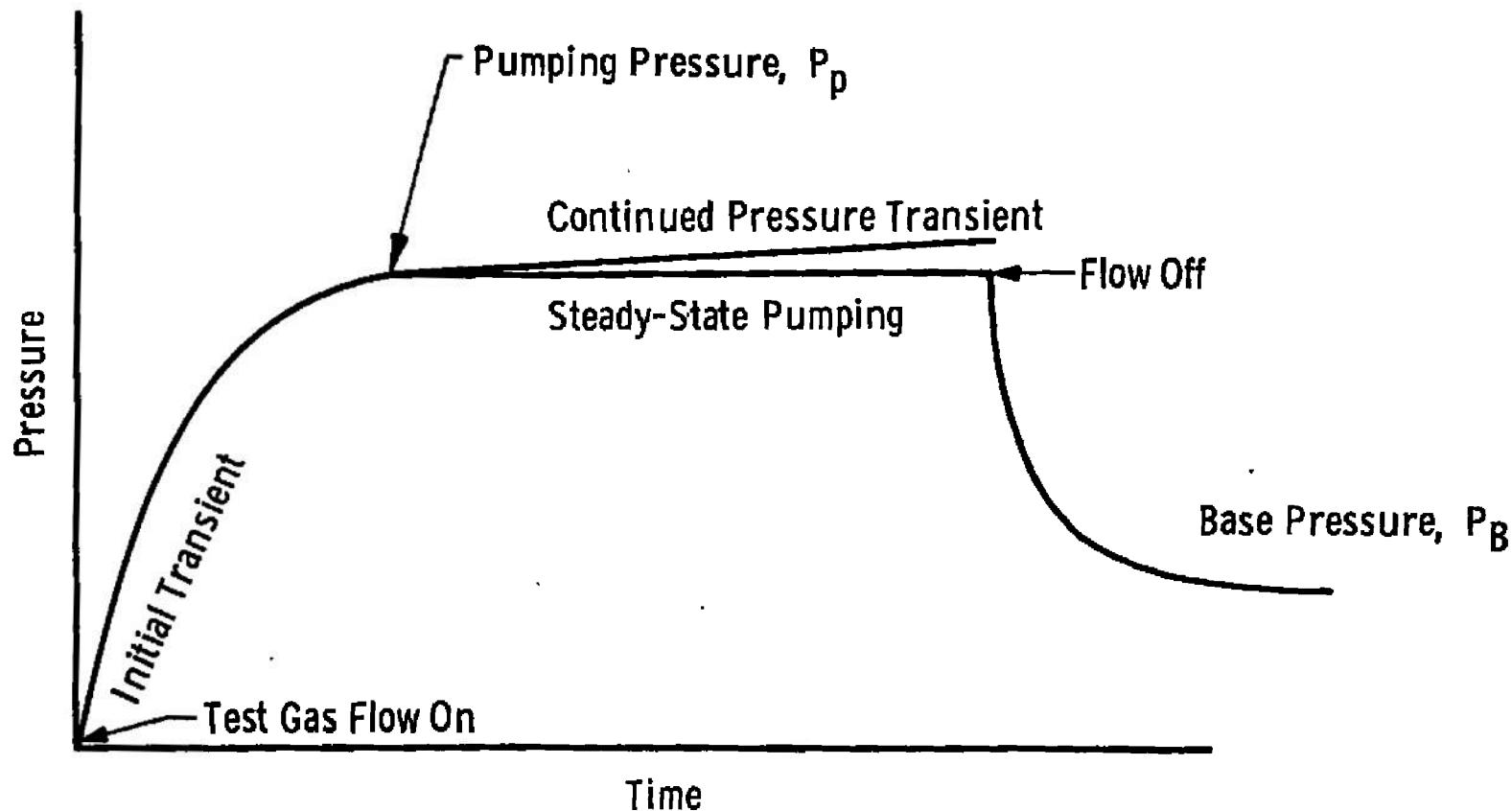


Fig. 8 Pressure-Time Cryosorption Data

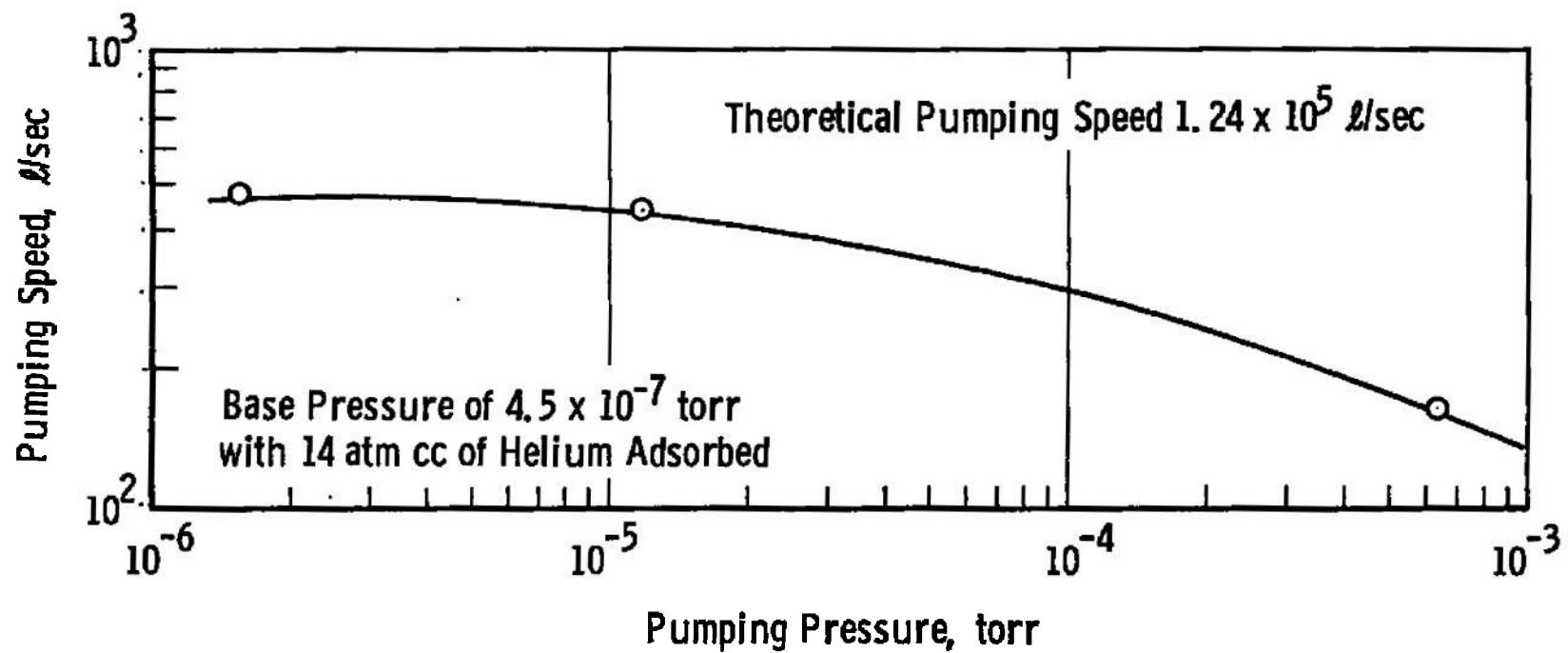


Fig. 9 77°K Helium Cryosorption Speeds on Molecular Sieve 5A, 13.6°K

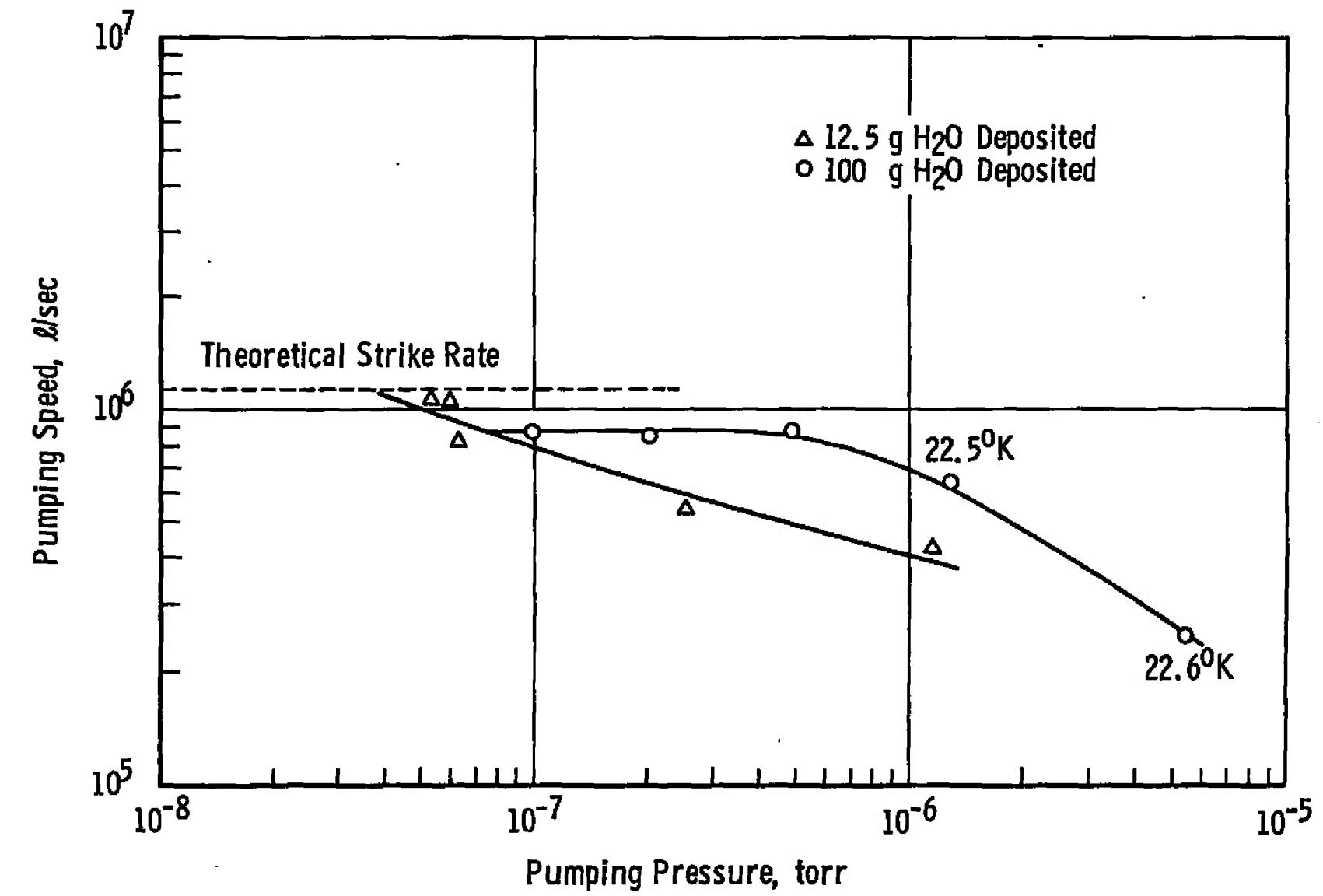


Fig. 10 Cryosorption Pumping of 300°K Hydrogen on 21.3°K Water Frost, 27.7- ft^2 Area

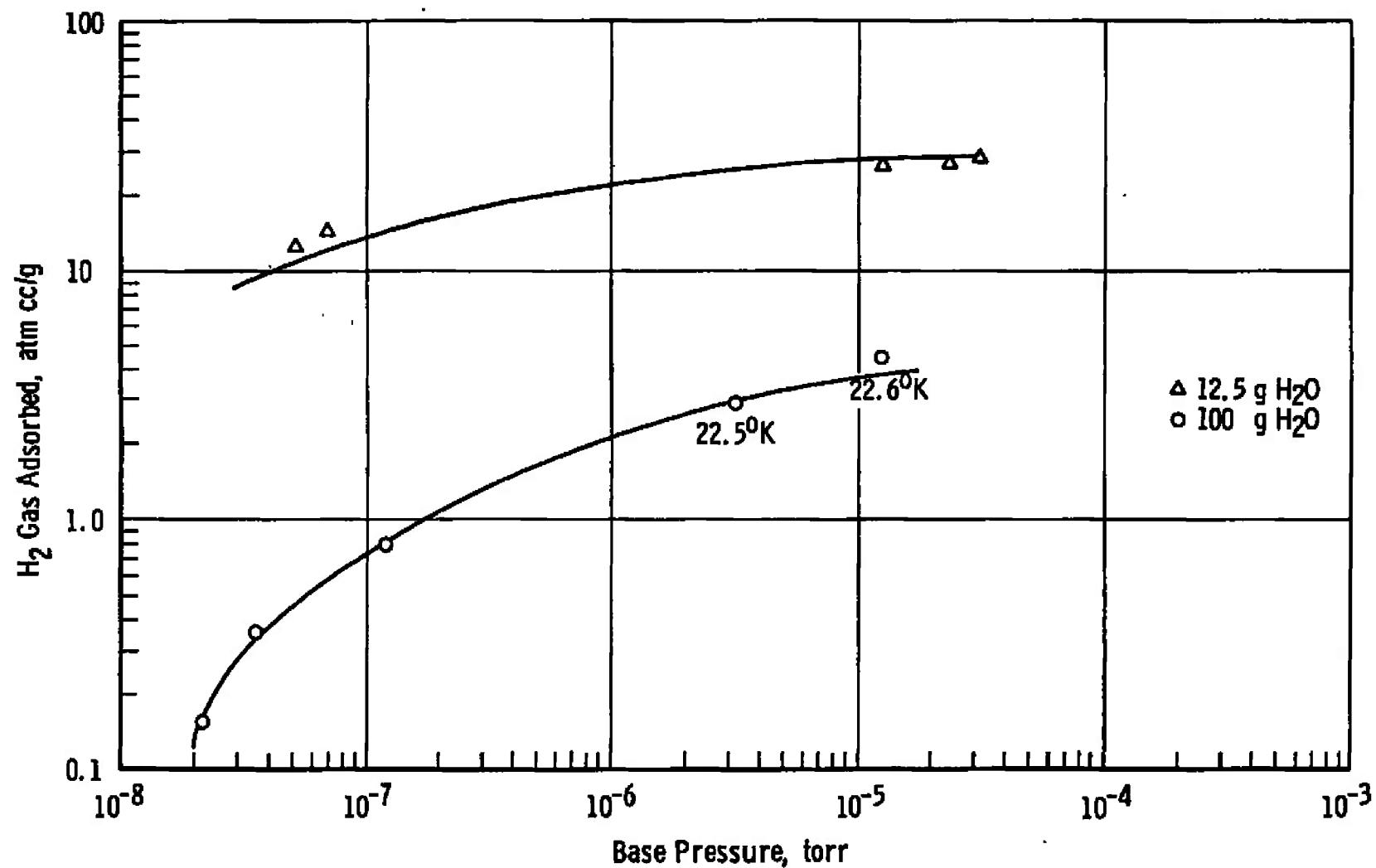


Fig. 11 Adsorption Isotherm for Hydrogen on Water at 21.3°K

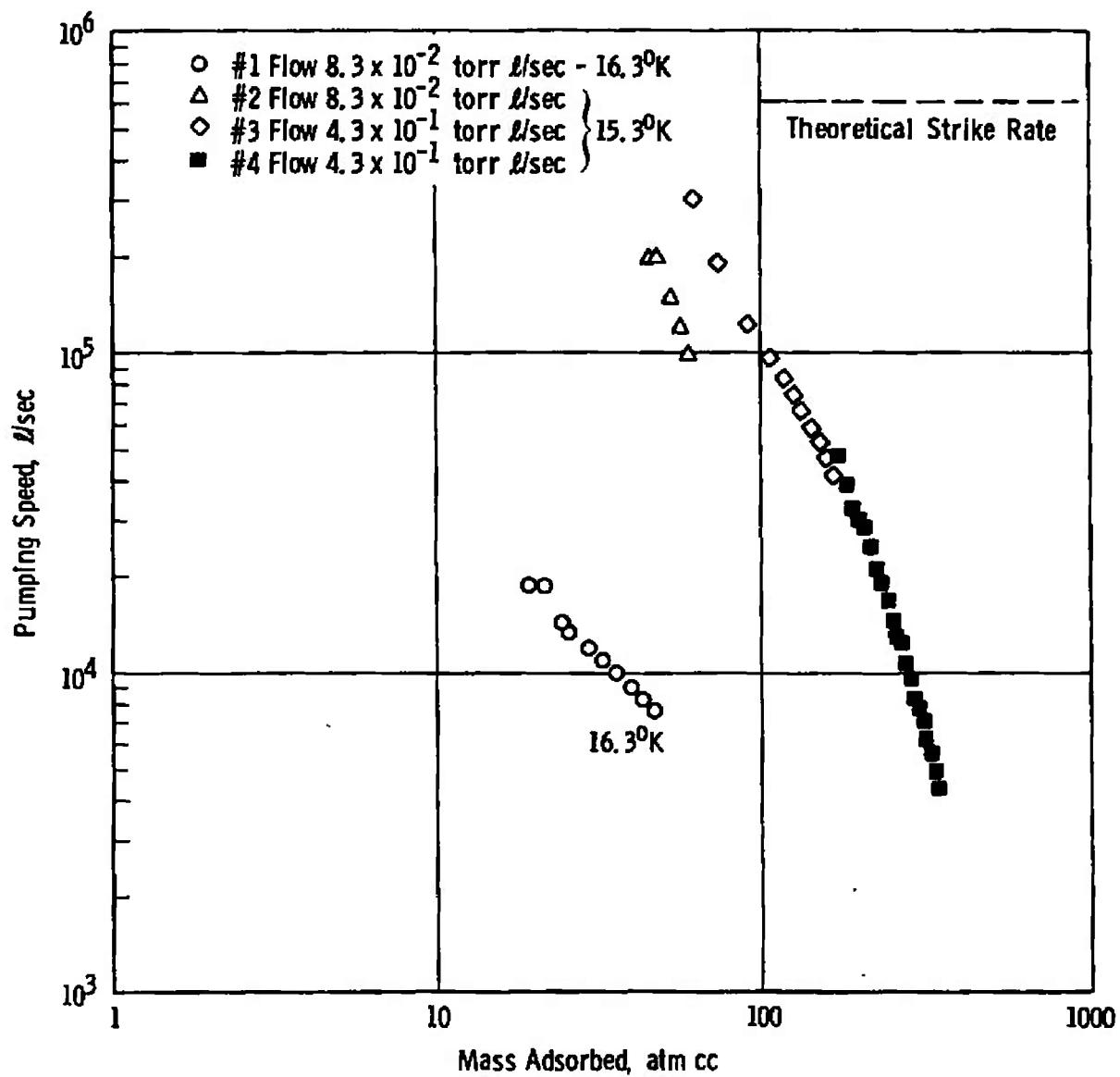


Fig. 12 Hydrogen Pumping Speed on Carbon Dioxide Frost versus Hydrogen Mass Preadsorbed

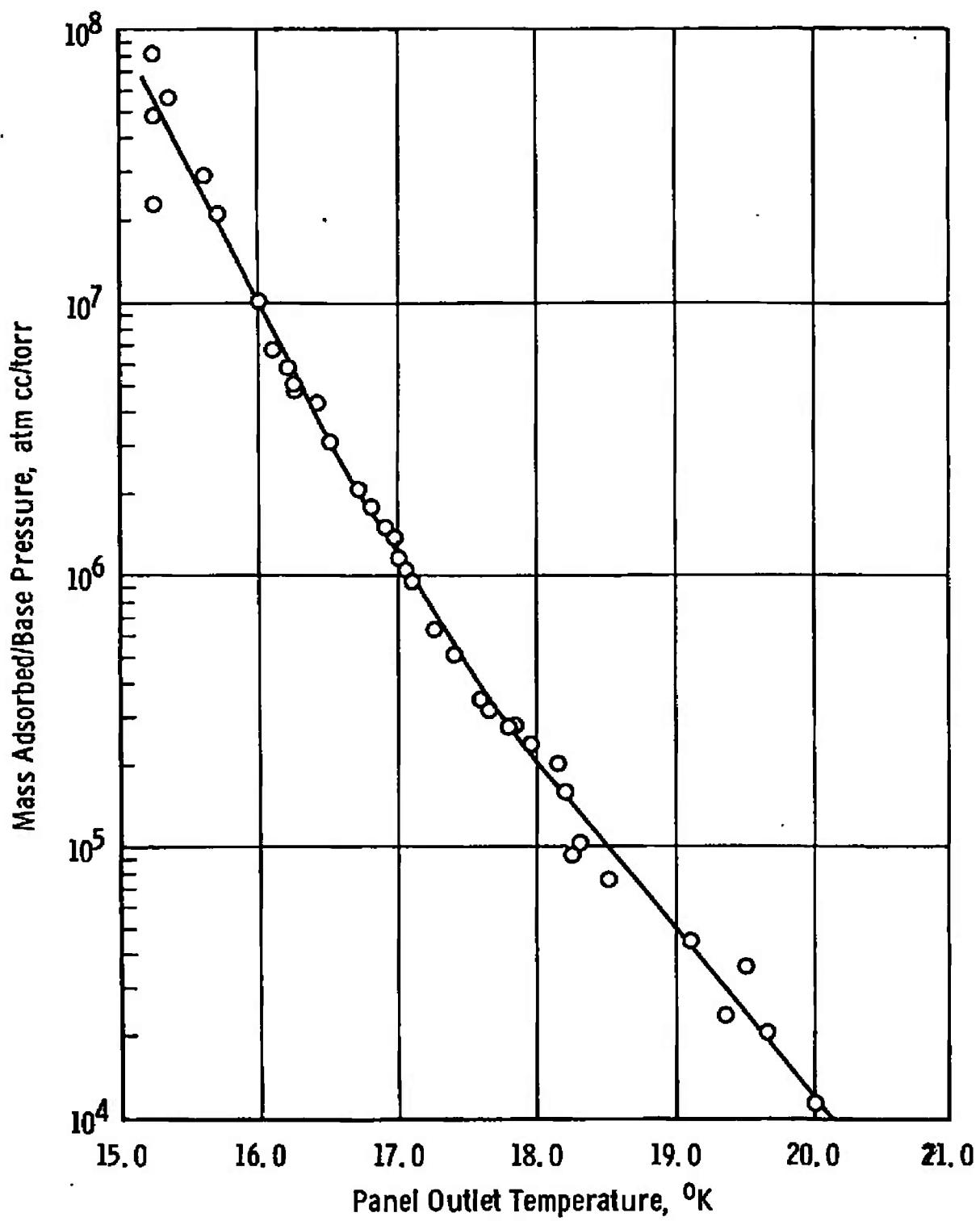


Fig. 13 Hydrogen Adsorbed per Base Pressure Ratio versus Panel Outlet Temperature;
Panel Coated with 22.4 grams Carbon Dioxide on 27.7- ft^2 Area

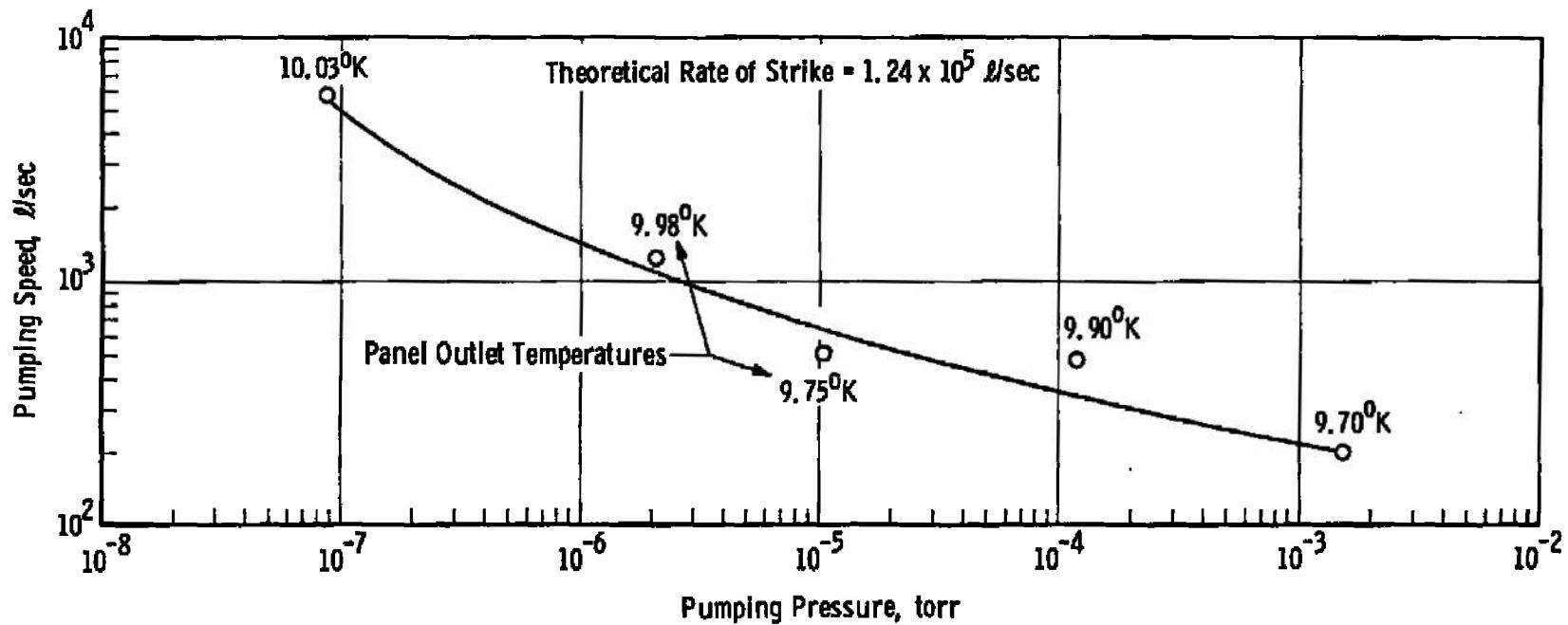


Fig. 14 Cryosorption Pumping of 77^0K Helium on Molecular Sieve 5A

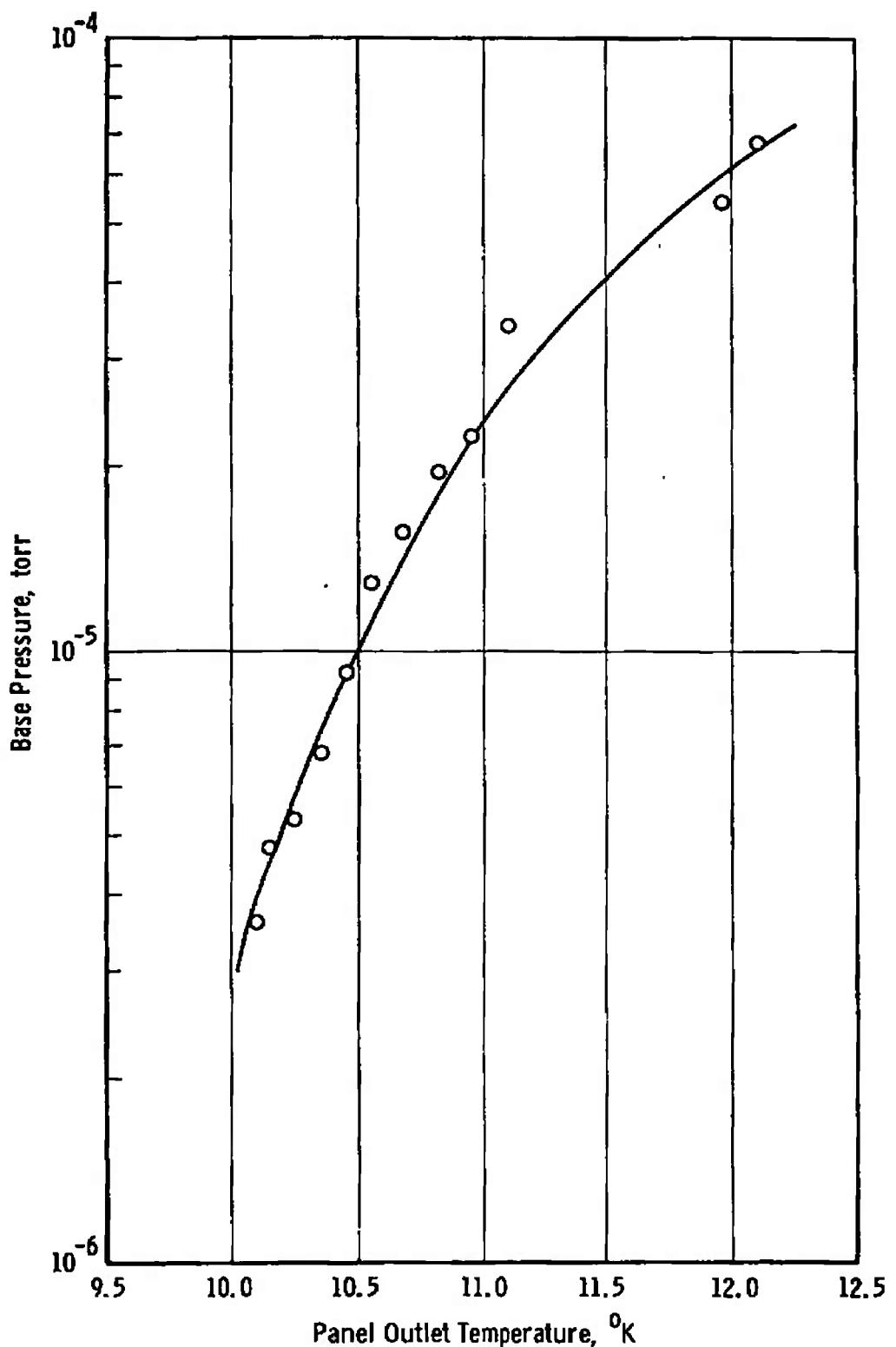


Fig. 15 Base Pressure versus Panel Outlet Temperature, Molecular Sieve 5A Coated,
8.02 atm cc Helium Adsorbed

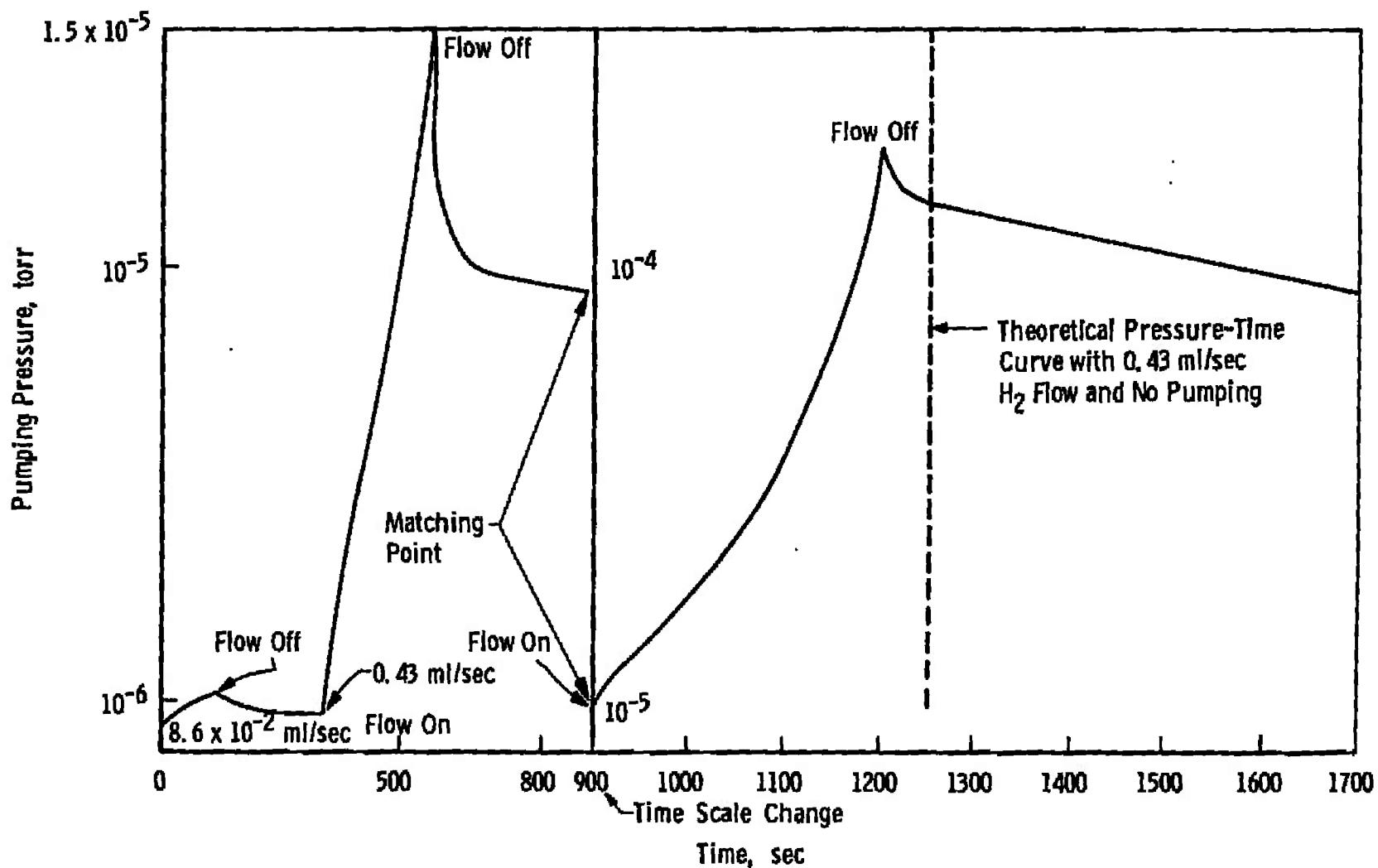


Fig. 16 Transient Pressure-Time Data, Hydrogen Adsorption on 15.3°K Carbon Dioxide Frost

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Experimental results are presented for helium adsorption on molecular sieve 5A at 10 and 13.6°K. Results are also given which show that water frost near 22°K adsorbs hydrogen at high volumetric rates with significant adsorptive capacity. Carbon dioxide frosts demonstrated these characteristics to a lesser degree near 16°K.

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